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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant : Davies, G.H. et al.
Serial No. : 09/936,794
Filed : November 13, 2001
For : Primer Coating of Steel
Group Art Unit : 1762
Examiner : Barr, Michael E.

DECLARATION UNDER 37 CFR 1.132

I, Peter Greenwood, do hereby declare as follows:

1. I am a citizen of Sweden residing at Vaktmästaregängen 11, Gothenburg. I am a product specialist at Colloidal Silica Group, Eka Chemicals AB, Sweden, where I have worked for 6 years.

I have been working with different applications of colloidal silica since 1992 in previous employments before my employment at Eka Chemicals AB. I have also worked with the preparation of high molar ratio silicates (molar ratio $\text{SiO}_2/\text{M}_2\text{O}$ between 4 and 5), prepared by mixing colloidal silica and silicate solution at a former employer, Nanosol AB, Sweden. I have a degree of MSc. in Chemical Engineering and a Licentiate degree in Engineering Chemistry (additional 2 years of research studies/work after the Master degree), both degrees at Chalmers University of Technology in Gothenburg.

2. U.S. Patent No. 3,977,888 (Sano)

Sano teaches, at Col. 3, lines 38-45, a silicate as a multi-component system comprising a mixture of monomeric silicate ions, polymeric silicate ion micelles, incomplete soluble amorphous silica or its



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hydrate, and amorphous silica with adsorbed silicate ions. The multi-component system is a highly viscous solution. The viscosity is in the range of 1000 to 15000 centipoises, see Col. 4, lines 56-59. Sano further discloses, at Col. 4, lines 60-61, that the amorphous silica powder used in the invention has a particle size of 0.5μ to 50μ . Sano also teaches that the amorphous silica filler is dissolved in the state of incomplete dissolution in the aqueous alkali silicate solution, Col. 3, line 52-55. Sano finally makes a distinction between the functions of the amorphous silica and the silicate solution; the silicate solution acts as a binder while the amorphous silica acts as filler, Col. 3, line 50-51. Sano does not describe any system comprising silica sols.

3. The difference between a silica sol and a silicate solution

The difference between a silica sol and a silicate solution can, in its simplest way, be explained by realizing that a silica sol is a stable colloidal dispersion of silica particles whereas a silicate solution is a solution of silicate ions.

A silicate solution normally contains semi-polymeric species like dimers, tetramers etc. The average molecular weight depends on the molar ratio between silica and alkali, typically the average molecular weight is in the order of 100 to about no more than 2000 grams per mole¹. The particle size of such species depends on the average molecular weight but is below about 2 nm, compare the molecular weight on for silicates given in page 133¹ with particle size table on page 191². The molar ratio $\text{SiO}_2/\text{M}_2\text{O}$ of a pourable concentrated silicate solution, i.e., a solution with an upper viscosity limit of about 10 000 – 20 000 centipoises, typically is below 4 – 5³; the maximal silica content is declining with an increase in ratio. The maximum silica content is about 35 per cent by weight at a ratio of 2.0-2.5 while at a ratio of about 4 the maximum silica content has decreased to about 25 per cent by weight³. Other parameters of such silicate solutions are given in table 2.1⁴.

A silica sol is a stable colloidal dispersion⁵ of silica particles, i.e., a dispersion in which the



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particles do not show any tendencies for settling. The reason for this is that the Brownian motion is larger than the sedimentation motion. This means that a dispersion that settles is not a colloidal system. In practice this means that the particle size range for a silica particle in a silica sol is in the region from about 5 to about 100-200 nm. Iler discloses that silica sols with particles of 300 nm (= 0.3 μ m) in particle size settles⁶. Silica sols are polymeric materials with average molecular weights of from about 50 000 -100 000 up to several millions grams per mole depending on particle size⁷. For silica sols, the molar ratio $\text{SiO}_2/\text{M}_2\text{O}$ is within the range of about 20 -1000, typically the ratio is in the range of 50 - 300.

Silicate solutions are normally used as raw material in the production processes of silica sols which are described in numerous patents^{8,9,10,11,12}.

Further, silica sols are low viscous products, typically the viscosity range is from about a few centipoises up to about 20 - 30 centipoises even for concentrated sols with a silica content of up to 50 % by weight¹², compared to silicate solutions that may have a viscosity of 1000 centipoises and above⁴ at a similar silica content.

References:

1. R.K. Iler, The Chemistry of Silica, p. 131-133, Wiley and Sons, 1979
2. R.K. Iler, The Chemistry of Silica, p. 191, Wiley and Sons, 1979
3. R.K. Iler, The Chemistry of Silica, p. 120, Wiley and Sons, 1979
4. R.K. Iler, The Chemistry of Silica, p. 119, Wiley and Sons, 1979
5. R.K. Iler, The Chemistry of Silica, p. 312, Wiley and Sons, 1979
6. R.K. Iler, The Chemistry of Silica, p. 313, Wiley and Sons, 1979
7. R.K. Iler, The Chemistry of Silica, p. 347, Wiley and Sons, 1979
8. U.S. Patent No. 2,631,134
9. U.S. Patent No. 2,650,200
10. U.S. Patent No. 2,270,345
11. U.S. Patent No. 3,012,973
12. U.S. Patent No. 3,673,104

I declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements



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were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent resulting therefrom.

DATE: June 7, 2004BY: Peter GreenwoodName: PETER GREENWOODTitle: Specialist